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Work supported during the period supported by grant NAG5-9422 over the course of its full duration (1999-2003) has contributed to 39 publications: 10 full-length publications and 29 extended abstracts (Appendix A). We also acknowledge the completion of the PhD thesis of Karl Kehm, who is now pursuing independent research in the laboratory of Dr. Jamie Gilmour, Manchester England. As usual, our collaborations have continued to play important roles in our research and we make an effort to open our laboratory to a wide variety of these efforts. Particularly productive over the past year are collaborations with Sasha Krot, Yuri Amelin, Jamie Gilmour, Gary Huss, Bob Nichols, Tom Bernatowicz, Charlie Kehm and Norbert Thonnard. This grant has therefore provided support for research in a wide diversity of fields.

This document, submitted as part of this proposal renewal represents the Final Report required by NASA for Grant NAG5-9442. It should be emphasized that, while this work statement in the original proposal outlined anticipated directions of our research, the specific activities we carried out during this period differed slightly from those proposed, capitalizing on new unexpected results and new advances in analytical capability. The thrust of all the work we completed were completely within the stated research goals of the proposal and significantly advanced our knowledge of planetary processes and our understanding of the early solar system. The following summary outlines our achievements in the different areas of research.

A. Early solar system processes and time scales using I-Xe chronometry: The I-Xe chronometer, based on the decay of 15.7 Ma ^{129}I, provides high-resolution records of post-formational meteorite evolution. Our group confirmed its validity by direct comparisons with Pb-Pb in separated minerals (Brazzle, et al., 1999). More recent work has shown that the I-Xe system is concordant with the Mg-Cr ages of St. Marguerite and Richardton feldspar (Polnau and Lugmair, 2001), with the Hf-W reported by Quitté, et al. (2000) and, most recently with Pb-Pb in individual Richardton chondrules and separated minerals (Pravdivtseva, *et al.,* 2002; Pravdivtseva, *et al.,* 1999). The I-Xe system, with its unique application to secondary metamorphism, can now be exploited to provide important new constraints on meteorite evolution (Hohenberg *et al.*, 2000; Pravdivtseva, *et al.,* 2000; Pravdivtseva and Hohenberg, 2000; Pravdivtseva, *et al.,* 2001; Pravdivtseva, *et al.,* 2002; Gilmour 1999).

1) Meteoritic magnetite and "Anomalous" I-Xe ages: The anomalously old I-Xe ages for Orgueil and Murchison magnetites reported by Lewis and Anders (1975) has long been an enigma since they apparently predated by 10 Ma the Pb-Pb ages reported for Allende CAIs, refractory inclusions that are generally acknowledged to be among the earliest objects formed in the solar system. Our comprehensive studies (Hohenberg, *et al.*, 2000) demonstrated those early results were in error, that Orgueil magnetite really formed at 4.563 Ga, postdating the Shallowater reference standard by 2.8 ± 0.3 Ma and Allende CAIs by nearly 6 Ma. Furthermore, we demonstrated that it was the failure of the KI irradiation monitor in the earlier work by Lewis and Anders (1975) that was the probable the cause of this error. Moreover, all I-Xe ages determined using KI, or those that use Murchison magnetite (MM, which was referenced to the KI monitor of the Lewis and Anders (1975) work) as the reference standard, are also in error, requiring a careful re-examination of many prior I-Xe results. We continued examinations of meteoritic magnetite, both processed with KCl solutions and the unprocessed magnetic fractions, confirming the results of Hohenberg, *et al.*, 2000 (Pravdivtseva and Hohenberg, 2001). Subsequent to this we obtained aliquots of the original Orgueil magnetite separates made by Roy Lewis (Lewis and Anders, 1975; Herzog *et al.*, 1973) which we then irradiated at the University of Missouri Research Reactor, measured the I-Xe ages and confirmed, for the third time, our earlier conclusions (Pravdivtseva and Hohenberg and Meshik, 2003). Recognition of calibration errors made by Lewis and Anders (1975) are very important because it removes the anomalously old ages of solar system magnetites, thought to be of secondary origin, adds new confirmation to I-Xe dating methods and brings the formation sequence of solar system material into an understandable order (Pravdivtseva, *et al.*, 2003). We obtained a collection of 13 CO meteorite samples from Sasha Krot and 2 CR samples from Tim Swindle. If magnetite is indeed of secondary origin in each of these, we may be able to resolve time differences in the alteration history on CI, CM, CO and CR parent bodies. This work is ongoing, under support of our subsequent grant.

2) Alteration history of the CV chondrites: One of the unique properties of the I-Xe chronometer is its sensitivity to post-formational processes due to the fact that most iodine hosts are secondary phases. The I-Xe isocron for sodalite, the major iodine carrier in Allende CAIs, indicates closure 3 Ma after Pb-Pb closure in the refractory phases, constraining the time of aqueous alteration on the CV parent body, with individual I-Xe ages of 3.0, 3.1 and 3.7 Ma after Shallowater (± 0.2) for 3 Allende CAIs (Hohenberg, *et al.*, 1997; 1999; Pravdivtseva and Hohenberg, 2001; Krot, *et al.*, 2002; Pravdivtseva, *et al.*, 2002). Allende dark inclusions, included in this study have individual I-Xe ages of -0.8 ±0.3, -1.1 ± 0.2, -1.5 ± 0.1 and -1.9 ± 0.2 *before* Shallowater (Krot, *et al.*, 2002; Pravdivtseva, *et al.*, 2002a,b). This seems to indicate two different stages of aqueous alteration, an early alteration stage, predating Shallowater by ~ 1 Ma, and a later stage terminating (lasting?) ~ 3 Ma after Shallowater. The I-Xe system of Allende dark inclusions were largely not reset by most recent alteration, but the fine-grained CAIs largely were (Pravdivtseva, *et al.*, 2002b,c; Pravdivtseva, *et al.*, 2003a,b). This fruitful effort continues in our subsequent NASA grant.

Also in collaboration with A. Krot, we have performed I-Xe studies on magnetite, pyroxene, feldspar and dolomite separates from the CV3 meteorites Bali, Groznaja, Kaba, Groznaja and Mokoya. Kaba and Bali magnetites yield well-defined isochrons 4.2 ± 0.3 and 7.9 ± 0.2 Ma, respectively, after Shallowater, compared with 2.8 ± 0.3 Ma for magnetites from the CI Orgueil (Pravdivtseva, *et al.*, 2001; Pravdivtseva and Hohenberg., 2001). The age differences are significant and, like those found with Allende CAIs, beyond experimental uncertainty, suggesting conditions for aqueous alteration on the parent body span several Ma. This work continues with the analysis of the other mineral fractions in a variety of different meteorites.

3) Comparision of I-Xe and Pb-Pb ages in the same object: Chondrules, minerals and the absolute calibration of I-Xe. In collaboration with Yu Amelin, we have undertaken an extensive comparison between Pb-Pb and I-Xe ages in chondrules, and later separated minerals, from common objects. This purpose of this work is two-fold: First and foremost an attempt to decipher formation histories, and to confirm common closure times for Xe and Pb systems. The second is to provide a better absolute calibration of I-Xe. Currently the only absolute I-Xe calibration is through the Pb-Pb age Acapulco phosphate (Nichols *et al.,* 1994; Brazzle *et al.,* 1999), but because the Pb-Pb system is somewhat disturbed, its age is less certain and less precise, and source of the 2 Ma uncertainties placed on all absolute I-Xe ages. Preliminary results (Pravdivtseva, *et al.,* 2002a,b) confirm this calibration (within the uncertainty), but suggest that absolute I-Xe ages may be too old by about 2 Ma. Attempts have been made to find objects with precise I-Xe and Pb-Pb ages to better normalize the I-Xe chronometer. Shallowater, has too little U (< 2ppb) to provide assistance, but the search continues.

Individual chondrules from Richardton, Elenovka, Bjurbole, Saratov, NWA267, Allende, and two un-named Antarctic LL3 meteorites have been irradiated for I-Xe, with portions saved for Pb-Pb studies. I-Xe and Pb-Pb measurements have already been made on 3 chondrules from Richardton (H5), the source of the comparisons indicated above (Pravdivtseva, *et al.*, 2002a,b). In Richardton chondrule #6, two release peaks and two distinct isochrons, differing in age by 2 Ma, were observed. The high temperature phase predates Shallowater by 2 Ma; the lower temperature isochron has the same age as Shallowater. We will use the laser probe on the remaining fraction of the chondrule in an attempt to locate the iodine hosts and better interpret this age difference. I-Xe studies of separated mineral components have just begun and will be a major focus of this work in the future.

Further work is now under way, in collaboration with Jamie Gilmour in Manchester, U.K., which will assimilate, evaluate and compare all available radionuclear ages to establish a refined record of early solar system evolution. This work has already demonstrated that the ages based upon the short-lived extinct radionuclides: ^{129}I-Xe, and ^{53}Mn-Cr and ^{26}Al-Mn are already self-consistent, providing enormous confidence in these systems while refining the absolute calibration of these chronometers (Gilmour, 1999), and this work continues under new grant support (Gilmour, Pravdivtseva and Hohenberg, in preparation).

4) The composition of trapped Xe in the early solar system: The I-Xe isochron represents a two component mixture between a single iodine-derived component and trapped Xe. In most cases, the trapped end member seems to be quite consistent with normal "planetary" Xe, represented by OC-Xe (Lavielle and Marti, 1992) or Q-Xe (Wieler *et al.,* 1992) but, in the case of Allende dark inclusions, trapped Xe seems to require a "sub-planetary" composition. That is, if the ^{128}Xe/^{130}Xe in the trapped component is set at the "planetary" value of ~ 0.083, the required ^{129}Xe/^{132}Xe ratio on that end member is below the "planetary" value of 1.04. This observation is unchanged if these isotopes are normalized to ^{130}Xe instead of ^{132}Xe. Although it is possible that trapped Xe can evolve in such a way that the ^{129}Xe/^{132}Xe ratio increases, for instance, in an environment of high iodine/Xe ratio in closed (parent body) systems (c.f. Kennedy *et al.,* 1988), there is no way to produce ^{129}Xe/^{132}Xe ratios that are *below* "planetary". This is because the solar nebula iodine/Xe ratio is about unity, and the ^{129}I/^{127}I ratio about 10^{-4}, restriction open system evolution of the ^{129}Xe/^{132}Xe ratio in the solar nebular to 10^{-4}. How then do we explain "sub-planetary" ^{129}Xe/^{132}Xe ratios? Swindle pointed out a trend in trapped compositions versus relative I-Xe age (Swindle, 1998), but many of the I-Xe isochrons were relatively imprecise and secondary features, such as trapped compositions, difficult to adequately constrain. New measurements in this laboratory confirm that "sub-planetary" compositions seem to be required, though understanding the source of these compositions remains the focus of much effort (Ozima *et al.*, 2002; Hohenberg *et al.*, 2002a; Hohenberg *et al.*, 2003a,b).

I-Xe isochrons from 4 Allende CAIs and 6 Allende dark inclusions show two clustered groups of alteration ages, ~ 4 Ma apart, and a range of trapped Xe compositions, each determined with more precision than in previous studies (Swindle, 1998). By comparing irradiated and unirradiated samples, we confirm that the $^{128}Xe/^{132}Xe$ ratios in the trapped components are identical to OC-Xe, but the isochrons often pass below OC-Xe, suggesting that the $^{129}Xe/^{132}Xe$ ratios are lower than OC-Xe. We propose that it may not be the ^{129}Xe that is anomalously low but the ^{128}Xe (in irradiated samples) that is anomalously high, an alternative to the nebular chemical evolution model (Ozima *et al.*, 2002). If ^{127}I is intimately mixed with trapped xenon, it can result in a trapped Xe pseudo-component with an elevated ^{128}Xe (after neutron irradiation). If this is true, it has important implications: a) similarly trapped, iodine can act much like an isotope of Xe, and b) trapping of iodine and Xe must have occurred late, after decay of most of the ^{129}I, placing new constraints on the duration of aqueous alteration processes. This problem remains a thorny one, and all of the visible solutions equally improbable. Understanding the source and evolution of meteoritic trapped Xe is an important and often overlooked issue that will have an impact on our understanding the primitive solar nebula. A definitive study of trapped Xe evolution in aqueously altered material is underway (Hohenberg, *et al.*, in preparation).

5) I-Xe dating and cooling rates of iron meteorites and chondrules: In collaboration with G. J. Wasserburg, we studied silicate inclusions from Colomera IIE iron. Using laser extraction from single diopside, feldspar and other mineral grains from Colomera (IIE), we determined that feldspar is the major iodine carrier of these phases. A composite feldspar sample of 12 individual mineral grains was irradiated yielding an absolute I-Xe isochron age of 4556 ± 2 Ma (Pravdivtseva, *et al*, 2001; Acapulco apatite normalization). Model I-Xe ages of individual temperature fractions of the Colomera feldspar approach the linear isochron age in the systematic manner of progressive closure. Using a) the laboratory extraction temperatures (1450-1550°C) or b) the closure temperatures derived the corresponding Arrhenius plots (420-730°C for sheet, cylinder or sphere models; E_{act} ~ 53 kcal/mol), age differences can provide cooling rates. Using the cooling theory (Dodson, 1973), our Colomera data yield a cooling rate of 2 to 4°C/Ma, and closure temperature ~ 450°C (Pravdivtseva, *et al.*, 2000; Pravdivtseva, *et al.*, 2001). Since only temperature *differences* are important, the derived rates are not that sensitive to actual closure temperatures. The factor of two range of derived cooling rates (2 to 4°C/Ma) encompasses the uncertainty of whether we take the actual laboratory extraction temperatures or the closure temperatures derived from the Arrhenius plots. This relative independence from knowledge of the actual closure temperature gives us confidence that we can obtain meaningful cooling history form the I-Xe structure. We plan to expand this work to include other irons, and to revisit previous data on the I-Xe system of Allende chondrules (Swindle *et al.*, 1983) in order to study more comprehensively the response of the I-Xe system to the relaxation of metamorphic conditions and the relation to specific cooling/alteration histories.

B. The Active Capture of Volatiles: A new mechanism for the capture of heavy noble gases, possible implications for phase Q and planetary heavy noble gases. Investigations of methods to capture volatiles during comet fly-through led to the discovery of a new process, active capture, for the incorporation of heavy noble gases into solids (Hohenberg, *et al.*, 1997; 1998; Meshik *et al.*, 2000). Our earlier work has demonstrated that heavy noble gases can be chemically bound to surfaces at active sites ("anomalous adsorption") much more firmly than by physical (normal) adsorption by Van der Waals forces. The active capture experiment for Stardust works when a low-Z metal is co-deposited along with impinging heavy noble gases whose energy is greater than a few eV. Capture efficiency of ~ 1 percent was observed (Hohenberg *et al.*, 2002a). This, oridinarily, would not be the case since the surface dwell time of these gases ($<10^{-11}$ sec, Kornelsen E. V., 1964) is far too short to allow them to be captured in a metal film which is being deposited at a rate of about one monolayer per .1 to .01 seconds. Something must be retarding the heavy noble gases so they stay on the surfaces long enough to be covered over. If the impinging atom has energy in excess of the surface work function (few eV), they can remove a surface electron, create a local vacancy and be chemically bound. There they can remain at the newly created active site long enough to be bound up in the growing metal matrix (Hohenberg, *et al.*, 2002a). "Anomalous adsorption" has been observed in the laboratory for some time and reflects the ability of heavy noble gases to be chemically, and sometimes quite stabily, bound to the surface (Garrison *et al.*, 1987; Niedermann and Eugster, 1992; Nuth *et al.*, 1987; Neimeyer and Leich, 1976). Active capture depends upon anomalous adsorption to work by providing an increase in surface dwell time that is sufficient for heavy noble gases to be permanently trapped by some other process. In lunar breccias and regolith (Drozd *et al.*, 1972; Behrmann *et al.*, 1973) anomalous adsorption bound "parentless" components (^{40}Ar, and Xe from ^{244}Pu and ^{129}I decay) long enough to be "fixed" by some other process (shock redistribution, agglutination, etc). It has often been a nuisance in the laboratory where the terrestrial atmosphere is mistaken identified as indigenous (Neimeyer and Leich, 1976; Niedermann and Eugster, 1992). Active surface sites can be produced by evaporation under ultra-high vacuum conditions, by fragmentation in vacuum, crushing or energetic particle irradiation. Such sites were certainly available in the primitive solar nebula, produced by the effects of ionizing irradiation (Nichols *et al.*, 1992; Feigelson *et al.*, 2002), or by the impinging solar wind itself (far greater than the work functions). The surface dwell times of heavy noble gases were certainly enhanced, greatly exceeding that of ordinary adsorption, and they could be bound permanently to these surfaces by a number of processes. Active capture can therefore provide a method to achieve the enormous (surface) concentrations of heavy noble gases, and it may provide a mechanism for the acquisition of planetary gases by phase-Q (Hohenberg, *et al.*, 2002a). This work, in collaboration with N. Thonnard, will continue with further exploration and more detailed study of the parameter space of this newly discovered process.

C. Separation of Xe-L from Xe-H: Physically selective experiments. We previously demonstrated that selective laser absorption is capable of separating Xe-H from Xe-L in

Murchison Cδ (Meshik *et al.,* 1998; 1999; 2000). In further study of this effect, and to eliminate the possibility that apparent separation may be an artifact of anomalous blank, Cδ separates were prepared from Indarch and Allende. These separates contain nearly pure Xe-HL, with 10 times less Xe-P3, providing laser targets for which trapped Xe (P3) plays less of a role. Results from this meteorite confirm the previous H-L separation by selective absorption, this time without the possibility of explanation by anomalous artifacts (Meshik, *et al.*, 2001a). We conclude that there are real, but subtle, physical differences in the host sites of Xe-H and Xe-L in Cδ with separation by selective optical absorption pointing toward different sub-populations which carry different proportions of these two constituents. More recent investigations involve other physical differences. Mobility of nanodiamonds in colloidal suspension depend upon surface properties, and these differences in these may also lead to differences grain populations, perhaps with detectable different Xe-H/L ratios. Results exploiting mobility differences under applied electric fields (electrophoresis) have, so far, yielded negative results (Meshik *et al.,* 2002) but these are only preliminary and work will continue in this area under the subsequent NASA grant.

D. Abundances of Presolar grains: In collaboration with Gary Huss (Arizona State University), we have an ongoing program to extend the database for abundances and noble-gas-component distributions in presolar diamond, silicon carbide, and graphite. During the past three years, we have gathered data for seven carbonaceous chondrites (Murray, Murchison, Axtell, Renazzo, Colony, Mokoia, and Acfer 214) and one ordinary chondrite (Adrar 003). Preliminary reports of these data have appeared in Huss et al. (2000, 2001, 2002a) and a paper summarizing the abundance data for carbonaceous chondrites and their implications for thermal processing in the solar nebula has been submitted to Meteoritics and Planetary Science (Huss et al., 2002b). This collaboration has been quite successful and we intend for it to continue. Work completed to date on a series of carbonaceous chondrites and showed that these objects are not closely related, as has previously been assumed (Huss et al, 2002a,b). For the upcoming part of this work, we will investigate ordinary and enstatite chondrites. We have already made measurements on the H3.2 chondrite RC075 and the L3.2 chondrite Adrar 003, and we will extend this data base to include another H3, two more L3's, and EL3 chondrite. Combined with data already in the literature (Huss and Lewis, 1995), these data should permit new insight into the relationships between chondrite classes.

E. Studies of Neon and Helium from single interstellar SiC and graphite grains: In collaboration with R. Nichols, measurements of exotic Ne and He on single interstellar SiC and graphite grains have now been completed (Nichols, *et al.,* 2002). This comprehensive effort reviews theoretical astrophysical production rates and accesses the implications for AGB, Wolf-Rayet, supernovae and nova sources for each of the individual grains.

F. Pre-compaction exposure of meteoritic grains and chondrules. The huge excesses of spallation-produced ^{21}Ne observed in some individual olivine grains from CM meteorites require pre-compaction exposure times of 150 to > 300 Ma in parent body regoliths if they were exposed only to contemporary sources of energetic particles (i.e.

galactic cosmic rays or solar flares, even at the *maximum* production rate by the secondary cascade, as shown in various publications from our group [c.f. Woolum and Hohenberg, 1993; Caffee *et al.*, 1987]). Now that I-Xe ages of sodalite, carbonates and other products of aqueous alteration on CM, CV and CI parent bodies (Hohenberg, *et al.*, 1999) have restricted compaction times to <10 Ma after CAI formation, more intense particle sources are required, the most likely being solar flares from a (naked) T-Tauri phase of the sun. Recent work by Feigelson *et al.*, (2002) have confirmed this conclusion, using out data, and have integrated it into a comprehensive astrophysical and observational context. Although the progress of this work has been dormant for a couple of years, due mostly to the lack of interesting new samples, Marc Caffee's move to Purdue has opened the door to a more active collaboration. We now have a new selection of individual olivine grains separated from chondrules, including Dhajala, Bjurbole, Parsa, Chainpur and Murchison (thanks to Goswami) to study pre-compaction exposure effects.

G. Geochemically Measured Half-Lives: Double beta-decay of Te and Ba isotopes. We previously reported the absolute $\beta\beta$-decay rates for ^{130}Te and ^{128}Te, the longest half-lives ever measured experimentally (Bernatowicz et al., 1993). Although the double beta decay of ^{130}Te is relatively easy to detect, the refinement of its half-life remains elusive, with results falling into two statistically distinct groups that differ by about a factor of two. Since the inferred half-life depends upon the Xe accumulation time, there is the potential problem of Xe loss. To address this, we irradiated four tellurium samples, producing ^{131}Xe from ^{130}Te n-capture. For all four samples, > 95% of the newly produced ^{131}Xe correlated in a one-to-one fashion with ^{130}Xe form double beta decay, implying quantitative retention (Meshik, *et al.*, 2001b,c; Meshik, *et al.*, 2002a,c). Although excluding the potential for gas loss, it does not address the question of whether the gas-retention age and Pb-Pb age (or age of the host rock) are the same. Other laboratories are similarly focused on the Te double beta decay enigma and work will continue in collaboration with Jamie Gilmour, Manchester, U.K.

Another extremely long weak-decay half-life has been measured geochemically: ^{130}Xe, the product of ^{130}Ba double K-capture (2EC), has been observed for the first time and its half-life measured to be $2.2 \pm 0.5 \times 10^{21}$ yr. In addition, we measured ^{132}Xe from weak decay of ^{132}Ba, setting a new lower limit for its half-life > $1.3 \pm 0.9 \times 10^{21}$ years (Meshik, *et al.*, 2001a,c). This is the culmination of a 6 year effort to find the best, highly shielded, sample of barite free of spallation-produced ^{130}Xe, with sufficient antiquity for accurate determination of the decay product.

H. Noble gases in stratospheric interplanetary dust particles. Noble gases in individual IDPs provide a record of both the original inventory and the effects of atmospheric heating (Kehm, *et al.*, 1998a,b; Kehm, *et al.*, 1999; Kehm *et al.*, 2002). Previous collaborations with K. Kehm (DTM), G. Flynn (SUNY) and S. Sutton (Univ. Chicago) to measure noble gases and trace elements in individual IDPs have allowed us to identify the thermal effects of entry, exposure ages, particles of unusual origin and constrained the SEP/SW ratio to be 10^{-2} to 10^{-3}, orders of magnitude greater than that deduced by direct measurement by spacecraft, but in line with values inferred from other meteorite studies (Kehm, *et al.*, 1999; Kehm, *et al.*, 2002).

I. New Analytical Instrument. We have obtained, in the NASA instrument development program SRLIDAP, funds to develop two new, multi-collector, ion-counting noble gas mass spectrometers. In addition, we are building, completely constructed in our own machine shop, an enhanced, multiple multiplier, noble gas mass spectrometer based upon our existing instrument we built in 1980 which established and has maintained current state-of-the-art in noble gas mass spectrometry (Hohenberg, 1980). Although these new instruments, all different in geometry, sensitivity, isotope resolution, and other capabilities, will be aimed at providing the optimum analytical capabilities for measuring the returned Genesis samples, they will also provide greatly enhanced measurement capabilities for our ongoing Cosmochemistry research program.

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